SLOVENSKI STANDARD
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Kemična analiza železovih zlitin - Analiza ferosilicija - Določevanje Si in Al v ferosiliciju s fluorescenčno spektrometrijo

Chemical analysis of ferrous materials - Analysis of ferro-silicon - Determination of Si and Al by X-ray fluorescence spectrometry

Chemische Analyse von Ferrolegierungen - Analyse von Ferrosilicium - Bestimmung von Si und Al in Ferrolegierungen durch Röntgenfluoreszenzanalyse

Analyse chimique des matériaux ferreux - Analyse du ferro-silicium - Détermination de Si et Al dans le ferro-silicium par spectrométrie de fluorescence de rayons X

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Chemical analysis of ferrous materials - Analysis of ferro-silicon - Determination of Si and Al by X-ray fluorescence spectrometry

Analyse chimique des matériaux ferreux - Analyse du ferro-silicium - Détermination de Si et Al dans le ferro-silicium par spectrométrie de fluorescence de rayons X

Chemische Analyse von Ferrolegierungen - Analyse von Ferrosilizium - Bestimmung von Si und Al in Ferrolegierungen durch Röntgenfluoreszenzanalyse

This Technical Report was approved by CEN on 24 April 2011. It has been drawn up by the Technical Committee ECISS/TC 102.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This document (CEN/TR 10354:2011) has been prepared by Technical Committee ECISS/TC 102 “Methods of chemical analysis for iron and steel”, the secretariat of which is held by SIS.

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1 Scope

This Technical Report describes a X-ray fluorescence (XRF) spectrometric method for the determination of Si and Al contents in ferro-silicon materials.

The method is applicable to:

— Si contents between 40 % and 90 %;

— Al contents between 0,5 % and 6 %.

The correction of the spectrometric measurement from spectral interferences on the analytical lines used is essential. This Technical Report is valid for the analytical lines:

— Si Kα 7.126 (for element contents between 45 % and 90 %);

— Al Kα 8.339 (for element contents between 0,8 % and 6 %);

— Fe Kα 1.937 (for element contents between 10 % and 58 %).

NOTE For matrix matching purposes, iron is included in the analytical program to be prepared.

Within the conditions here above, spectral interferences don't need to be calculated.

2 Principle

Preparation of oxide beads, comprising the oxidation of the sample with strontium nitrate and its melting with lithium tetraborate, in a platinum crucible.

The beads are irradiated by an X-ray beam of suitable energy. The secondary X-rays produced are dispersed by means of crystals and the corresponding intensities are measured by detectors at the selected wavelengths.

The contents of the relevant elements are determined by relating the measured intensities of unknown samples to calibration curves recorded with beads prepared with certified reference materials.

Fixed channel or sequential systems may be used to provide simultaneous or sequential determinations of element concentrations.

3 Reagents

During the analysis, use only reagents of recognised analytical grade.

3.1 Strontium Nitrate [$Sr(NO_3)_2$]

3.2 Sodium Carbonate [Na_2CO_3]

3.3 Lithium Tetraborate [$Li_2B_4O_7$]

3.4 Sodium Iodide [NaI]

4 Apparatus

4.1 X-ray fluorescence spectrometer

The spectrometer can be either a simultaneous or a sequential wavelength dispersive model and shall be optimised according to the manufacturer instructions.

4.2 Melting devices

4.3 Platinum crucibles

4.4 Nickel crucibles

4.5 Platinum dishes

5 Safety precautions

They shall be in accordance with national regulations for X-ray equipment.

X-ray equipment shall be used only under the guidance and supervision of a responsible, qualified person.

6 Sampling

Sampling shall be carried out in accordance with appropriate international or national standards for ferro alloys.

7 Procedure

7.1 Test sample preparation

In a platinum crucible (4.3), weigh 6,00 g of $Li_2B_4O_7$ (3.3) to the nearest 0,001 g.

In a nickel crucible (4.4), weigh, to the nearest 0,001 g:

— 0,750 g of $Sr(NO_3)_2$ (3.1);

— 2,500 g of Na_2CO_3 (3.2);

— 1,000 g of $Li_2B_4O_7$ (3.3).

Then, weigh 0,250 0 g of the sample, to the nearest 0,000 1 g and add it into the same nickel crucible (4.4).

Finally transfer into the nickel crucible (4.4) 0,020 g of NaI (3.4), weighted to the nearest 0,001 g.

Carefully mix all the components added into the nickel crucible (4.4) and then completely pour the mixture contained in the nickel crucible (4.4) into the platinum crucible (4.3), onto the 6,00 g of $Li_2B_4O_7$ (3.3), whilst avoiding the contact of the mixture with the wall of the platinum crucible (4.3).

Introduce the platinum crucible (4.3) in a furnace set up at the temperature of 650 °C for 6 min, in order to oxidise the sample.

Melt the sample at the temperature of 1 200 °C, whilst mixing continuously and then pour the melted sample in a platinum dish (4.5) preheated with a Bunsen flame.

Allow the so prepared bead to cool.

7.2 Preparation of the calibration curves

Prepare the calibration of the X-ray fluorescence spectrometer by using oxide beads of ferro-silicon Certified Reference Materials (CRMs).

For each element, prepare a minimum of six oxide beads with different contents of Si, Al and Fe, in the same way as for the test samples (see 7.1), only using Certified Reference Materials. These Certified Reference Materials shall cover the entire calibration range.

Use the analytical line Kα 7.126 for Si, Kα 8.339 for Al and Kα 1.937 for Fe and plot the absolute intensities versus the related certified contents.

NOTE The calibration curves can also be obtained by using oxide beads prepared with pure metals and/or oxides. Annex C describes the related preparation.

7.3 Spectrometric measurements

7.3.1 Spectrometric measurements of the calibration and the re-calibration samples

For each element (each analytical line) measure the related absolute intensity of the calibration samples together with the re-calibration samples according to the instrument manufacturer instructions, at least four times each.

NOTE 1 To compensate the instrumental day to day drift, re-calibration samples and procedures are required.

NOTE 2 It's advisable to prepare the re-calibration samples as described in 7.1. These samples can be internal reference materials provided that their homogeneity has been carefully check, approved and recorded.

Prepare the related calibration graphs by plotting the mean intensity values of the Certified Reference Materials beads against the corresponding Si and Al certified contents.

Record the regression calculations and store the relevant parameters, together with the corresponding coefficients of correlation, the standard error of estimate, the error of the slope and the confidence limits.

Check the calibration trueness by measuring a set of Certified Reference Materials not used in the calibration and prepared as described in 7.1. The silicon and aluminium certified values of these samples shall cover at least the low, mid and top points of the corresponding calibration ranges.

7.3.2 Spectrometric measurements of the test samples

Following the manufacturer instructions, measure the intensity of each test sample bead at least twice and calculate the mean value of the corresponding results.

8 Expression of results

On the calibration curves, read the contents, expressed in percent (mass fraction) of the elements (Si, Al and Fe) determined in the test sample.

9 Precision

Four laboratories in two European countries participated in an inter laboratory test programme under the auspices of ECISS/TC 102/WG 5, involving three determinations of Al and Si at several levels.

Each laboratory carried out two determinations under repeatability conditions as defined in ISO 5725-1, i.e. one operator, same apparatus, identical operating conditions, same calibration and a minimum period of time. The third determination was carried out on a different day using the same apparatus with a different calibration.

Details on the samples used are given in Annex A and the results obtained are reported in Annex B.

The sets of data available were too few for a statistical evaluation based on ISO 5725-2 and ISO 5725-3. Only Mandel's h and k statistics were used (see Figures B.1 to B.4) rather for illustrative purposes.

A short comment on the suitability of the data presented is also given in Annex B.

10 Test report

The test report shall contain the following information:

a) identification of the test sample;

b) method used;

c) results;

d) any unusual characteristics noted during the determination;

e) any operation not included in this Technical Report or in the document to which reference is made or regarded as optional;

f) date of the test and/or date of preparation or signature of the test report;

g) signature of the responsible person.

Annex A
(informative)

Test samples used for the precision test

The test samples used are listed in Table A.1, here below. Elements and/or contents reported in grey cells were out of the scope of the precision test.

Table A.1 — Composition of the test samples used for the precision test

Sample label	Al (%)	Ti (%)	P (%)	Si (%)	Fe (%)	B (%)
NBS 59 A	0,35		0,016	48,1	50,0	0,058
DL 2307	6,01	0,073	0,020	55,9	12,6	
NBS 58 A	0,95	0,051	0,009	73,2	25,2	0,001
IPT 56	0,31	0,020	0,022	75,0	24,1	
ECRM 582-2	1,15	0,225	0,018	75,2	21,4	0,005
JSS 720-4	1,52		0,032	76,4		
ECRM 529-1	0,86	0,090	0,032	91,1	6,2	